

Original Research

Impact behaviors of poly-lactic acid based biocomposite reinforced with unidirectional high-strength magnesium alloy wires

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Abstract

A novel poly-lactic acid (PLA) based biocomposite reinforced with unidirectional high-strength magnesium alloy (Mg-alloy) wires for bone fracture fixation was fabricated by hot-compressing process. The macroscopical and microscopical impact behaviors of the biocomposite were investigated using impact experiments and finite element method (FEM), respectively. The results indicated that the biocomposite had favorable impact properties due to the plastic deformation behavior of Mg-alloy wires during impact process. While the content of Mg-alloy wires reached 20 vol%, the impact strength of the composite could achieve 93.4 kJ/m², which is approximate 16 times larger than that of pure PLA fabricated by the same process. According to FEM simulation results, the complete destruction life of the composites during impact process increased with increasing volume fraction of Mg-alloy wires, indicating a high impact-bearing ability of the composite for bone fracture fixation. Simultaneously, the energy absorbed by Mg-alloy wires in the composites had a corresponding increase. In addition, it denoted that the impact properties of the composites are sensitive to the initial properties of the matrix material.

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Keywords: Impact property; Magnesium alloy wire; Poly-lactic acid; Composite; FEM

1. Introduction

Impact loading against bone is a common phenomenon in day life, as it happens when we do running, jumping, weight lifting, etc. Most of these impact loadings are beneficial for maintaining bone mass and structure [1,2], while some powerful and incident impacts, however, could result in bone fracture due to the limited impact resistance of bone and osteoporosis according to the increase of age [3,4]. Then, orthopedic immobilization is applied using bone fracture fixation devices. As the impact is unavoidable during healing, the impact properties of these devices, also concerned as the ability of resistance against an impact are significant.

Most of traditional metallic materials for bone fracture fixation devices have excellent impact properties. However, the non-degradation and probable toxic ions release from these materials

are still the strategies [5–10]. Recently, poly-lactic acid (PLA) based bio-polymer attracted huge interests due to its biodegradation, versatility and biocompatibility [11–16]. Unfortunately, the impact properties of most investigated PLA were poor, and they could not sufficiently meet the requirements of human bones. Thereby, many researches tried to overcome this shortcoming [12–14]. A high-strength composite composed with poly-L-lactic acid (PLLA) with high molecular weight as matrix and hydroxylapatite (HA) as reinforcement was fabricated [13]. Its impact strength could reach 166 kJ/m² when HA content is 30 wt%. Wan et al. [14] developed a unidirectional continuous carbon fiber-reinforced PLA composite with an excellent tensile-resistant performance, but its impact strength was only 25 kJ/m² with a high content (40 vol%) of carbon fiber. Additionally, it should be mentioned that most of these researches on the impact properties are macroscopical, while the microscopical studies such as the impact behavior of each component in the composite during the impact have not been analyzed quantitatively.

In this paper, a novel PLA-based biocomposite unidirectionally reinforced with high-strength magnesium alloy wires for

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bone fracture fixation was fabricated. The macroscopical impact behavior of this composite was studied through experimental impact tests and fracture morphologies investigation, while the microscopical impact behavior was simulated by finite element method (FEM). The aims of this work were to deeply understand the impact behaviors of the composite, and furthermore provide scientific basis for the design of this composite.

2. Materials and methods

2.1. Materials

AZ31B magnesium alloy wires with a diameter of 0.3 mm were utilized in this research, which were fabricated through a continuous process including smelting, casting, hot-extrusion, wet-drawing and annealing.

PLA with a low viscosity-average molecular weight of 60,000 g/mol was purchased from Shenzhen Esun Industrial Co., Ltd., China. Its crystallinity, glass transition temperature and density was 38%, 64 °C and 1.24 g/cm³, respectively.

The tensile properties of magnesium alloy (Mg-alloy) wires and PLA are illustrated in Table 1 and Table 2. The wires have an excellent deforming performance, as the value of its tensile strain is as large as 16%, while their tensile strength is 316 MPa. PLA exhibits an elastic deformation, as its tensile strain and tensile strength is 1.15% and 46 MPa, respectively.

2.2. Fabrication of the composites

The composites were fabricated with a lamina-stacked method. First, thin composite laminas were produced through pouring PLA/chloroform solution to overlay unidirectional Mg-alloy wires and subsequently evaporating in the air. Then several composite laminas were stacked symmetrically in a mold cavity. Thereafter, hot-compression process was applied to compress the stack under the condition of the temperature of 190 °C and the pressure of 5 MPa. Lastly, the samples were free cooled down to room temperature under the pressure of 5 MPa. Different composites with 5, 10 and 20 vol%

Mg-alloy wires were prepared, respectively and unreinforced PLA underwent the same fabrication process was compared as controls.

2.3. Characterization

2.3.1. Impact properties

Unnotched impact samples with a dimension of 60 mm × 12 mm × 2 mm, as shown in Fig. 1(a), were applied to evaluate impact energy by ZBC impact testing machine. The impact strength (σ_i) was calculated using the following equation:

$$\sigma_i = \frac{W}{bh}, \quad (1)$$

where W is the absorbed energy of the composite (J), b is the width of the sample (mm), and h is the thickness of the sample (mm).

2.3.2. Surface morphology

A Philips XL30 FEG SEM was used to characterize the fracture surface morphologies with an accelerating voltage of 22 kV. Before SEM, fracture surfaces of the samples were sputtered with a gold layer in an argon atmosphere.

2.4. Finite element method (FEM)

Finite element method (FEM), also referred as finite element analysis (FEA), was applied to stimulate the impact process. The material parameters are illustrated in Tables 1 and 2, and the impact model is depicted in Fig. 1(b). During simulation, the impact head and supporters were regarded as the rigid body with a density of 7.8 g/cm³, while beam and solid element type was assigned for Mg-wires and PLA, respectively. A tension and shear-based failure criteria was applied for both Mg-alloy wires and PLA, while Lagrange multipliers were used for interaction coupling [17]. Meanwhile, an hourglass control method based on the formulation of Belytschko and Tsay [18] was utilized to inhibit hourglass modes and a mesh erosion technique was used to remove distorted elements. The initial velocity of impact head was 5.24 m/s and the span was 40 mm, which were the same as that in practice.

3. Results

3.1. Impact characterizations acquired by experimental tests

Fig. 2 shows the change of the impact strength of the composites with the volume fraction of Mg-alloy wires. Apparently, the impact strength increases significantly in proportion to the volume fraction of Mg-alloy wires. The impact strength of pure-PLA was only 5.5 kJ/m², while the impact strength of the composite with 5 vol% Mg-alloy wires was 17.8 kJ/m², about twice larger than that of pure-PLA. As the content of Mg-alloy wires increases, the impact strength of the composite further achieves 93.4 kJ/m² at 20 vol% Mg-alloy wires. This amazing increase sufficiently reflected an excellent resistance ability of the composites against the

Table 1
Tensile properties of Mg-alloy wires (ϕ 0.3 mm).

| Poisson ratio | Density (g/cm ³) | Elastic modulus (GPa) | Elastic strength (MPa) | Tensile strength (MPa) | Tensile strain (%) |
|---------------|------------------------------|-----------------------|------------------------|------------------------|--------------------|
| 0.35 | 1.8 | 45 | 270 | 316 | 16 |

Table 2
Tensile properties of poly-lactic acid (PLA).

| Poisson ratio | Density (g/cm ³) | Elastic modulus (GPa) | Tensile strength (MPa) | Tensile strain (%) |
|---------------|------------------------------|-----------------------|------------------------|--------------------|
| 0.36 | 1.24 | 4.0 | 46 | 1.15 |

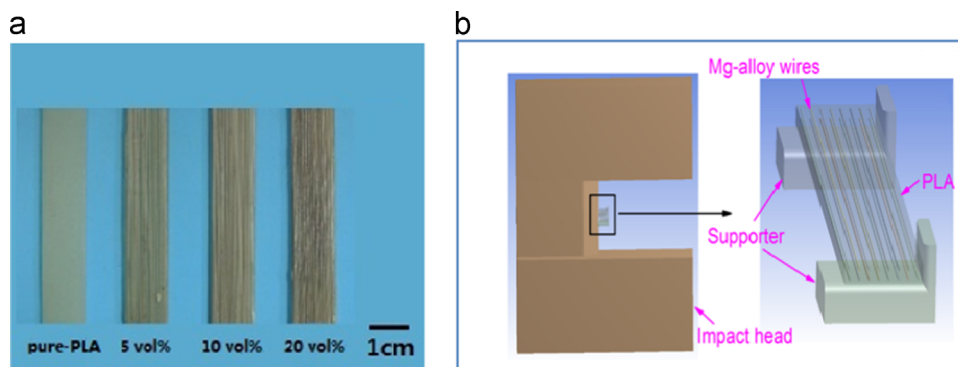


Fig. 1. Samples with different contents of Mg-alloy wires for impact tests (a) and their model for FEM (b).

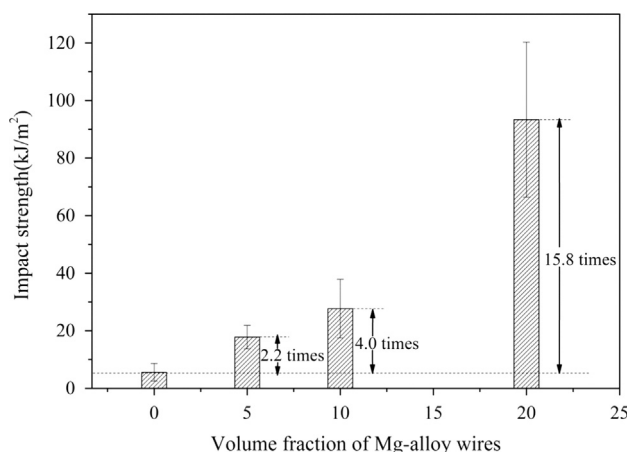


Fig. 2. Changes of the impact strength of the composites with the volume fraction of Mg-alloy wires.

impact. It is noticed that the impact strength of the composites did not increase linearly with the volume fraction of Mg-wires, but in an exponential-like model, indicating a complex reinforcing mechanism.

Furthermore, SEM was used to characterize the impact fracture morphologies of the composites, as shown in Fig. 3. Surface cavities where the wires fractured were observed. At 10 vol% Mg-alloy wires, an obviously outstretched wire was viewed, as pointed by the red arrow. Moreover, the diameter of this wire at the section seemed to have a significant decrease, indicating a plastic deformation occurred before fracture. Plenty of river pattern cracks around Mg-alloy wires were noticed. It seemed these cracks originated from the interface between Mg-alloy wires and PLA matrix, and then propagated in the matrix. It should be mentioned that the propagation of these cracks was an energy consumption process which could consume enormous energy during impact. Thereafter, the surface cavities were further investigated under higher magnification times. A decrease in the diameter and a great amount of dimples at the fracture section of wires were clearly observed. These characteristics further provided more evidences to verify the plastic deformation of wires during impact, which may probably lead to the apparent increase of the impact strength of the composites compared with pure-PLA.

3.2. Impact characterizations simulated by FEM

The impact test is a kind of macroscopical analysis method. Through the test, we could acquire the energy absorption of the composite during impact, as well as the impact strength. However, the microscopical quantitative results, such as the energy absorption of each component which was essential to further understand impact behaviors, could not be acquired. Thus, FEM, as a kind of time-costless and accurate method [19–22], was used to determine the microscopical impact behaviors.

Fig. 4 depicts the internal energy histories of the composite with 5 vol% Mg-alloy wires and its components during the impact process. The internal energy here could be concerned as absorbed energy. It is confirmed that the histories could be divided into three stages: the first stage was the moving of impact head to contact with the sample. Followed then was the second stage where impact happened between impact head and the sample, and the time horizon of this stage was defined as impact period in this paper. The last stage was the time increased to the predetermined calculating time as the impact already accomplished in the second stage. The curves of internal energy of the composite and its components have a similar tendency, exhibiting an exponential-like increase with the time. According to the curves, the impact period and impact strength of the composites could be easily acquired.

Then, the impact strength of the composites corresponding to the different volume fraction of Mg-alloy wires were gained, as represented in Fig. 5(a). The impact strength of the composites calculated by FEM is appropriate with the experimental results, though a slight decrease is observed. This slight decrease may probably derive from the condition of FEM, which ignored the air resistance and mechanical abrasion. That is to say, the FEM results are believable and they may truly reflect the impact resistance of the composites. Moreover, the impact periods of the composites were investigated, as seen in Fig. 5(b). Obviously, the impact period of the composite has a proportional increase with the content of Mg-alloy wires. For pure-PLA, it was only 0.0017 s, while it increased to 0.00191 s in the composite with 5 vol% Mg-alloy wires. As the element sizes of wires and PLA in these composite were the same, changes of the impact period could, then, directly reflect

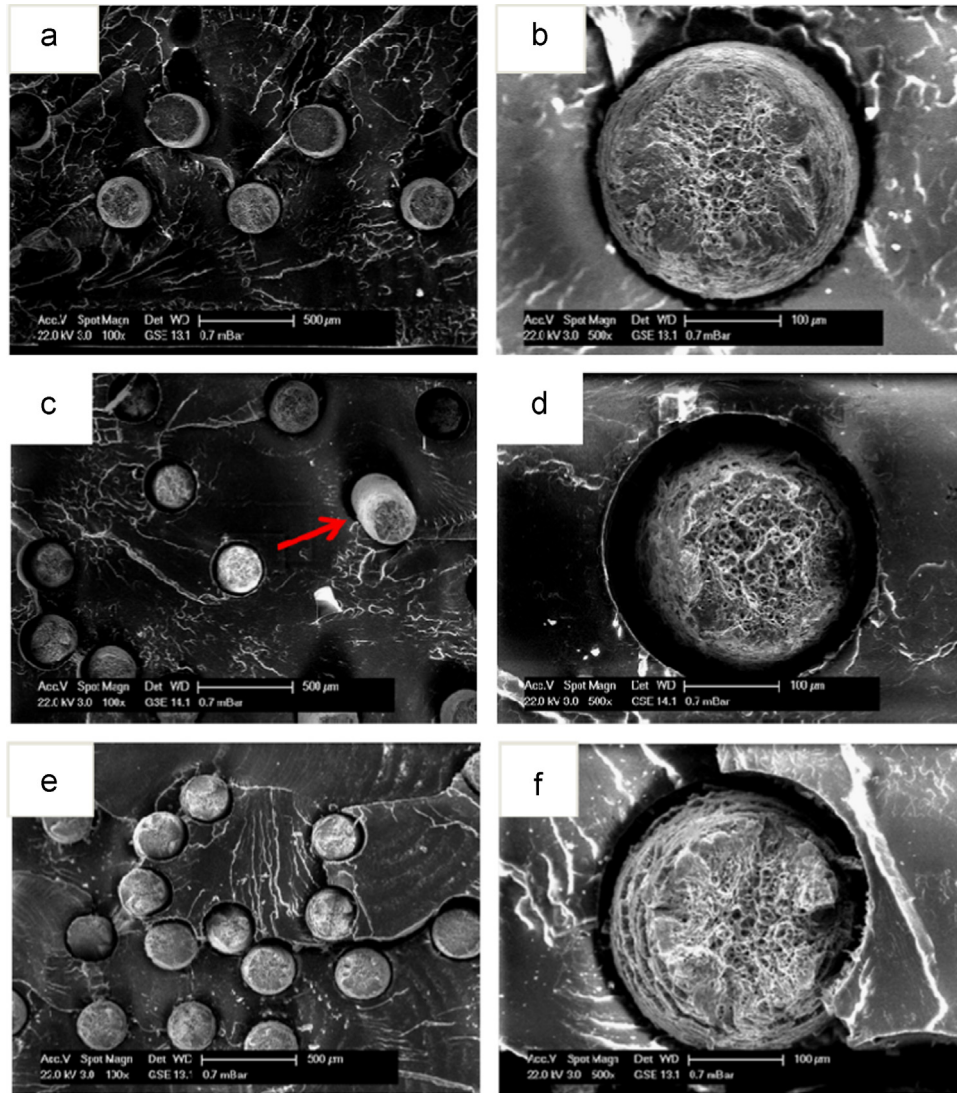


Fig. 3. Impact fracture morphologies of the composites with different volume fraction of Mg-alloy wires: (a) 5 vol%, low magnification; (b) 5 vol%, high magnification; (c) 10 vol%, low magnification; (d) 10 vol%, high magnification; (e) 20 vol%, low magnification; (f) 20 vol%, high magnification. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

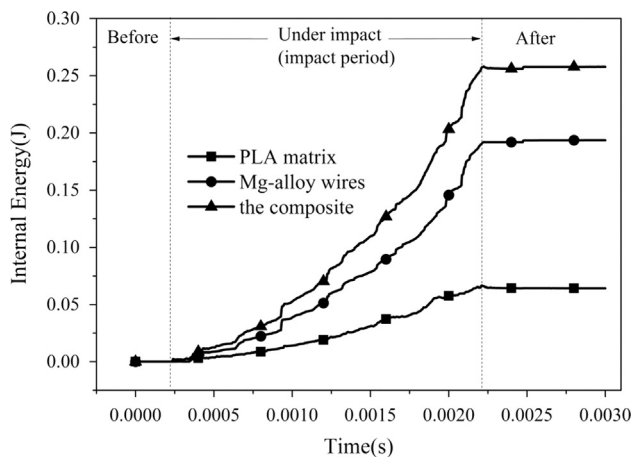


Fig. 4. Morphologies and internal energy histories of the composite with 5 vol% Mg-alloy wires and its components during the impact process.

changes of time needed for the complete destruction of the composite in practice. Apparently, a higher value of the impact period indicates a better impact bearing ability of the composite. At 20 vol% Mg-alloy wires, the value of the impact period could reach 0.00268 s, about 0.58 times larger than that of pure-PLA. This favorable value suggested a high level of impact resistance of the composite at 20 vol% Mg-alloy wires.

In addition, the internal energy of Mg-alloy wires in the composites was also studied, as shown in Fig. 6. At 5 vol% Mg-alloy wires, the internal energy of Mg-alloy wires was 0.192 J, while that of PLA matrix was 0.066 J, much less than that of pure-PLA, which was about 0.111 J. This energy decrease of PLA was probably original from its content decrease. At 20 vol% Mg-alloy wires, though the composite had a lower content of PLA, the internal energy of PLA matrix, however, exhibited a confused increase, reaching about

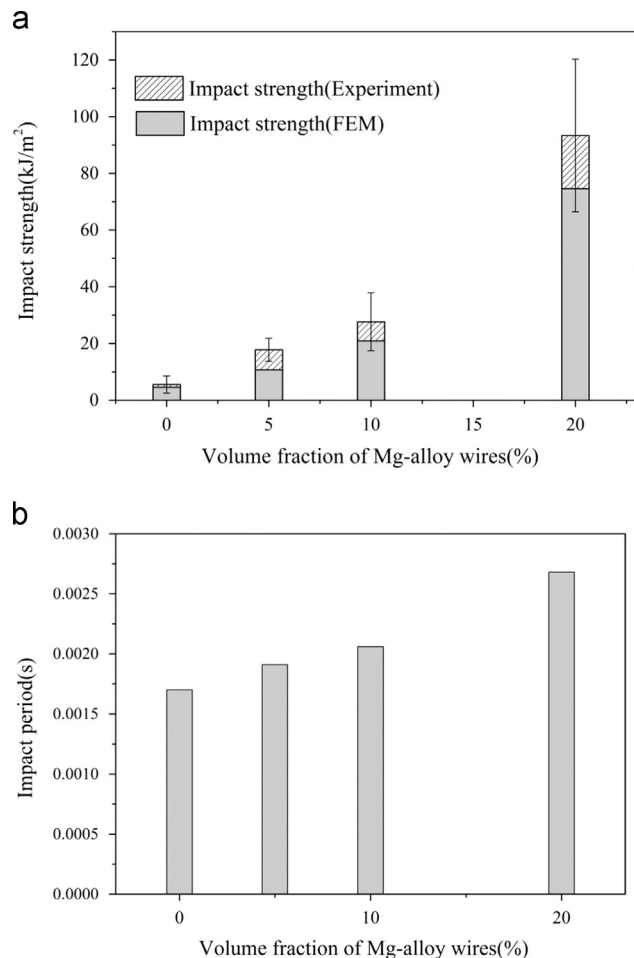


Fig. 5. Impact strength (a) and impact periods (b) of the composites with different volume fractions of Mg-alloy wires acquired by FEM.

0.28 J. Simultaneously, the internal energy of Mg-alloy wires had a dramatic increase, reaching ab out 1.51 J. Moreover, it seemed the percentage of internal energy of Mg-alloy wires to the composite had a logarithm-like increase in proportion to the content of Mg-alloy wires. At 5 vol% Mg-alloy wires, the percentage was nearly 75%, demonstrating Mg-alloy wires were already the main energy absorber, though the content was very low. It is interesting to notice that the percentage increased slowly from 83.7% at 10 vol% to 84.4% at 20 vol%. This much slower increase of the percentage compared with the content suggested there may be a maximum value of the percentage. Furthermore, the average internal energy per wire in the composite was calculated, and the results are illustrated in Table 3. At 5 vol% and 10 vol% Mg-alloy wires, the average internal energy per wire was approximately 0.012 J, while it increased significantly to 0.022 J at 20 vol%, indicating Mg-alloy wires played a complex reinforcing role on the impact properties of the composites.

4. Discussion

Bone fraction fixation devices are widely used to fix the fracture bones, which usually result from an incident and

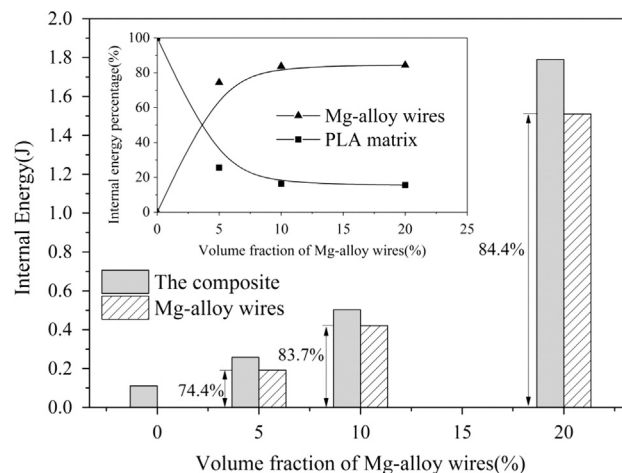


Fig. 6. Changes of the internal energy of Mg-alloy wires in the composites with different wire contents.

Table 3

Average internal energy per wire in the composites.

| Volume fraction of Mg-wires in the composites (%) | 5 | 10 | 20 |
|---------------------------------------------------|-------|-------|-------|
| Average internal energy per wire (J) | 0.012 | 0.012 | 0.022 |

powerful impact. The commonly used material for fabricating these devices could be divided into two aspects [5–15], including non-degradable metal and its alloys as traditional materials, while bio-polymer and bio-degradable metal as recently developed materials. However, these materials more or less have disadvantages that limited their applications.

In this research, we developed a novel bone fracture fixation material. This material is a kind of PLA-based composite material unidirectionally reinforced with magnesium alloy wires. The reinforcements in this material have a high tensile strength, as well as an excellent plastic deformation, which could sufficiently improve the mechanical properties of the matrix. Moreover, it seems the reinforcements could stabilize the pH value and further reduce the risk of delayed adverse biological response of the tissues which correlated with the acidic degradation products of the PLA matrix [23–25]. Additionally, it should be mentioned that this composite exhibited more excellent performance against a transient and powerful impact compared with pure-PLA, showing a bright application foreground in orthopedics.

4.1. Macroscopical impact behavior of the composites

PLA utilized in this research is a kind of biodegradable polymer with scarcely plasticity. It has extremely lower molecular weight than that used in other similar researches [13,14], while its elastic modulus and tensile strain is only 4.0 GPa and 1.15%, respectively. These excessively poor properties may lead to the poor performance of pure-PLA against a transient and powerful impact. Unlike PLA, Mg-alloy wires used in this study have excellent mechanical properties, while its tensile strain could achieve 16%, nearly 14 times

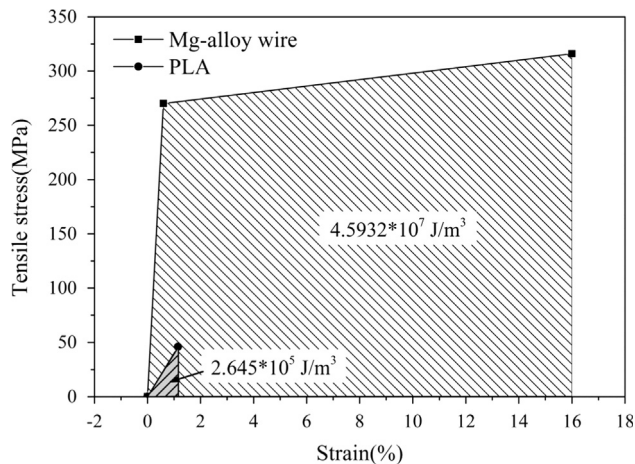


Fig. 7. Simulated tensile curves of Mg-alloy wires and PLA in FEM.

larger than that of PLA. This greater value of the wires indicates an excellent strengthening effect on PLA matrix, and moreover, the unidirectional arrangement could fully underplay this strengthening against the impact.

It is well known that the area under the tensile curves is proportional to the work required to deform a sample until it fails. Namely, the value of this area is equal to the value of the work to fracture per volume of the sample, defined as toughness, which is roughly proportional to impact strength. Then, the toughness of Mg-alloy wire is $4.5932 \times 10^7 \text{ J/m}^3$, while $2.645 \times 10^5 \text{ J/m}^3$ for PLA, as shown in Fig. 7. Apparently, the value of the toughness of Mg-alloy wire, referred as the energy to fracture per volume, is much larger than that of PLA. According to the surface morphologies of the fracture of the composites, Mg-alloy wires occurred obviously plastic deformation during impact process, indicating huge energy absorption of Mg-alloy wires, due to their high toughness, and furthermore, demonstrating the obvious increase of impact strength of the composites in proportion to the content of Mg-alloy wires. In addition, it should be mentioned that though carbon fibers used in other research had much larger tensile strength and tensile modulus which were 2000 MPa and 196 GPa, respectively, than that of Mg-alloy wires used in this study, the toughness of the carbon fiber, however, had a lower value, about $1.0204 \times 10^7 \text{ J/m}^3$ [14]. This lower toughness further resulted in relatively poorer impact performance of the composites. The impact strength of the composite at a high content of 40 vol% carbon fibers was only about 25 kJ/m^2 , while that was 93.4 kJ/m^2 for the composite at 20 vol% Mg-alloy wires in this study. As we know, the impact strength of bones varies from ages and parts, such as the impact strength of the femora could vary from 4 to 70 kJ/m^2 versus ages [26]. The requirements on the impact properties are different. As the impact strength of the composite in this study varies with the content of Mg-alloy wires, this versatility further may probably meet the different requirements of the bones.

It should be denoted that though the toughness of PLA was relative low, it also played an important role on the impact resistance, as it intermediary transfers the force to Mg-alloy

wires. This force could result in the appearance of cracks at the interface between matrix and reinforcements, due to the significant difference in the rigidity of the components. Moreover, these cracks could propagate in the matrix, and further consumed plenty of energy.

4.2. Microscopical impact behavior of the composites

It is interesting to notice that the impact strength of the composites did not increase linearly with the content of Mg-alloy wires. This phenomenon may indicate a kind of complicate reinforcing mechanism of Mg-alloy wires. Then, the microscopical impact behavior of the composites was studied.

The impact test in this study was a dynamic and transient process, and the external impact energy was about 260 J, much larger than the work to fracture of the composites. Meanwhile, the high external energy demonstrated that the destruction of the composite was instantaneous, and the impact periods of the composites calculated by FEM verified this version. The impact period of pure-PLA was 0.0017 s, and the value of 0.0017 s was so little that it could not be obviously detected. As a larger value of impact period means more time needed for the complete destruction of the composite, then the relative high value of 0.00268 s at 20 vol% Mg-alloy wires directly reflected the high level of impact resistance of the composite, which was greatly attributed to the reinforcement of Mg-alloy wires, as well as the increase of interface area between PLA matrix and Mg-alloy wires. In practice, this interface somehow played a significant role in shock wave dissipation and dispersion, and could further slowdown the shock propagation in the composites [27].

Furthermore, the energy absorption of each component in the composites was studied. It was surprised to realize that at a low content of 5 vol% Mg-alloy wires, the percentage of the energy absorption of Mg-alloy wires occupied 74.4% of that of the composite. With the content of Mg-alloy wires increasing, the energy absorption of Mg-alloys and PLA matrix both increased. It is easy to understand the energy absorption increase of Mg-alloys as their content increase, while the energy absorption increase of PLA matrix is somehow confused, as the content of PLA matrix decreases. This surprised increase of PLA may derive from the increase of interface between PLA and Mg-alloys, where the cracks and friction appeared [28,29]. Additionally, it seemed the percentage of the energy absorbed by Mg-alloy wires had a limit value, as it increased so slighter compared with the content of Mg-alloy wires increase. Namely, it means the percentage of the energy absorption of PLA matrix may have a minimal value, as shown in Fig. 6. That is to say, if the energy absorbed by PLA increases 1%, compared with the same increase percentage of Mg-alloy wires, the increase value of total energy absorption of the composite would be times larger. This result well confirmed the importance of the mechanical properties of matrix on improving energy absorption capacity of the composites, meeting well with the results in other research [30], where suggested that the matrix material should have a

greater strain at failure than the fiber to obtain the maximum energy absorption from a particular fiber.

Apparently, the average energy of wires per volume could be easily calculated, and the value was $5.19 \times 10^6 \text{ J/m}^3$ at 20 vol% Mg-alloy wires, while that was approximately $2.83 \times 10^6 \text{ J/m}^3$ at 5 vol% and 10 vol\% Mg-alloy wires. Accordingly, the average energy of PLA per volume was 7.7×10^4 , 4.82×10^4 , 6.32×10^4 and $2.43 \times 10^5 \text{ J/m}^3$, corresponding to the content of Mg-alloy wires at 0, 5, 10 and 20 vol%, respectively. These changes explained the nonlinear change of impact strength of the composites versus the content of Mg-alloy wires. In addition, these values are much less than that of their toughness under tension, indicating further improvement on the impact properties of the composites.

5. Conclusion

Macroscopical and microscopical impact behaviors of the novel magnesium alloy wires unidirectionally-reinforced PLA composites were investigated. The results indicate that this kind of composites has an excellent versatility of impact strength which could meet different requirements of bones, while its great impact properties are attributed to the plastic performance of the Mg-alloy wires during impact process. FEM results show Mg-alloy wires are already the main impact bearing supporter of the composites at a relative low content of 5 vol% Mg-alloy wires. Changes of the percentage of energy absorption of PLA matrix to the composite demonstrate that the initial mechanical properties of PLA play an important role on the impact strength of the composites. Moreover, a long time needed for the complete destruction of the composites suggests these composites could provide a great impact-bearing ability for bone fracture fixation.

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